

PTO 09-0935

CC=JP
DATE=20020308
KIND=A
PN=0269623

CO-CR-PT-B-GROUP TARGETS AND MAGNETIC RECORDING MEDIUM
[CO-CR-PT-B-KEI TAAGETTO OYABI JIKI KIROKU BAITAI]

TOMONORI UENO

UNITED STATES PATENT AND TRADEMARK OFFICE
WASHINGTON, D.C. NOVEMBER 2008
TRANSLATED BY: SCHREIBER TRANSLATION, INC.

PUBLICATION COUNTRY (10) : JP

DOCUMENT NUMBER (11) : 0269623

DOCUMENT KIND (12) : A

PUBLICATION DATE (43) : 20020308

APPLICATION NUMBER (21) : 2000260372

APPLICATION DATE (22) : 20000830

INTERNATIONAL CLASSIFICATION (51) : C 23 C 14/34 ; C 22
C 19/07 ; G 11 B
5/64 ; 5/851 ; H 01 F
10/16

PRIORITY COUNTRY (33) :

PRIORITY NUMBER (31) :

PRIORITY DATE (32) :

INVENTOR(S) (72) : UENO, TOMONORI;
MURATA, HIDEO;
TANIGUCHI, SHIGERU

APPLICANT(S) (71) : HITACHI METALS LTD.

DESIGNATED CONTRACTING STATES (81) :

TITLE (54) : CO-CR-PT-B-GROUP
TARGETS AND
MAGNETIC RECORDING
MEDIUM

FOREIGN TITLE [54A] : CO-CR-PT-B-KEI
TAAGETTO OYABI JIKI
KIROKU BAITAI

[Claim]

[Claim 1]

A Co-Cr-Pt-B-group target wherein the mean diameter of a cell split by a network formed by a boride is 200 μm and under.

[Claim 2]

A Co-Cr-Pt-B-group target as described in Claim 1 wherein the mean crystal grain size of the matrix is 40 μm and under;

[Claim 3]

A Co-Cr-Pt-B-group target as described in Claim 1 and Claim 2 wherein the dispersions in the mean diameter of a cell split by a network formed of a boride are 0.5 to 2.0 at a target surface part mean diameter as/target center part mean diameter aM ;

[Claim 4]

A Co-Cr-Pt-B-group target as described in Claim 1 through Claim 3 wherein the dispersions in the mean crystal particle size of the matrix are 0.5 to 2.0 at target surface part mean particle size bs /target center part mean particle size bm ;

[Claim 5]

A Co-Cr-Pt-B-group target as described in Claim 1 through Claim 4 wherein $1 \leq B \leq$ at %, $5 \leq Pt \leq 30$ at %, $10 \leq Cr \leq 30$ at %, and the residue is essentially made up of Co;

[Claim 6]

A Co-Cr-Pt-B-group target as described in Claim 1 through Claim 5 wherein $0 < (Ti + Zr + Hf + V + Nb + Ta + Mo + W + Mn + Re + Ru + Os + Rh + Ir + Ni + Pd + Cu + Ag + Au + C) < 40$ at %;

[Claim 7]

A magnetic recording medium having at least one or more layers made up of a Co-Cr-Pt-B-group thin film which is formed using the targets indicated in any of Claims 1 through 6 on a non-magnetic substrate.

[Detailed Description of Invention]

[0001]

[Technical Field]

The present invention relates to a Co-Cr-Pt-B-group target and a magnetic recording medium used to form a magnetic film for a magnetic recording medium used for magnetic disk devices and the like.

[0002]

[Prior Art]

In the prior art, Co-group magnetic films which were capable of high-density magnetic recording were developed and Ta and Pt were added to the Co-group magnetic film. In addition, B is added to Co-group magnetic films so that it has been reported that the magnetic properties were significantly improved (Journal of Applied Physique, 84, 6202 (1998) and the like.

[0003]

Methods used to produce this Co-group magnetic film include the sputtering method and other methods as described in the abovementioned publications. A target which is a source of supplying the film composition is required in the sputtering method. A target used to form a Co-group magnetic film to which B above has been added is made by casting in a cylindrical iron mold and the resulting cast clumps are sliced to a prescribed thickness.

[0004]

[Problems Which the Present Invention is Intended to Solve]

The inventors studied targets used to form Co-group magnetic films such as those described in the

abovementioned Journal of Applied Physics 84, 6202 (1998).

As a result, there were problems in that there were dispersions in magnetic properties such as the holding power and the angle type ratio of the magnetic film produced.

[0005]

The cast target is made of relatively fine chill crystals, coarse columnar crystals which depend on the cooling direction and relatively coarse equiaxed crystals so that the crystals were coarse and uneven. This caused dispersions in magnetic properties such as holding power and angle type ratio of the magnetic film. It is an object of the present invention to provide a Co-Cr-Pt-B-group target having a fine uniform texture.

[0006]

[Means Used to Solve the Problems]

The inventors found that the problem of dispersions occurring in the properties of the magnetic film when the Co-Cr-Pt-B-group cast target produced by slicing the resulting cast ingots to a prescribed thickness were a function of the cell split by a network formed by a boride,

that is, the size of the dendrite and they achieved the present invention.

[0007]

This means that the present invention is a Co-Cr-Pt-B-group target wherein the cell split by the network formed by a boride has a mean diameter of 200 μm and under.

[0008]

The Co-Cr-Pt-B-group target in the present invention is such that the mean crystal particle size of the matrix is 40 μm and under.

[0009]

In addition, the Co-Cr-Pt-B-group target in the present invention is such that the dispersions in the mean diameter of the cell split by the network formed by a boride should be 0.5 to 2.0 at target surface part mean diameter as (hereinafter, "as") / target center part mean diameter aM (hereinafter, " aM ") and the dispersions in the mean crystal particle size of the matrix is 0.5 to 2.0 at target surface part mean particle size bs (hereinafter, "bs") / target center part mean particle size bM (hereinafter "bM").

[0010]

The preferred composition of the Co-Cr-Pt-B-group target in the present invention may be $1 \leq B \leq 15$ at %, $5 \leq Pt \leq 30$ at %, $10 \leq Cr \leq 30$ at %, with the residue essentially made up of Co. Further, one, two or more types of element selected from Ti, Zr, Hf, V, Nb, Ta, Mo, W, Mn, Re, Ru, Os, Rh, Ir, Ni, Pd, Cu, Ag, Au and C may be $0 \leq (Ti + Zr + Hf + V + Nb + Ta + Mo + W + Mn + Re + Ru + Os + Rh + Ir + Ni + Pb + Cu + Ag + Au + C) \leq 40$ at %.

/3

[0011]

In addition, a magnetic recording medium can be manufactured stably by forming a Co-Cr-Pt-B-group magnetic film using the Co-Cr-Pt-B-group target in the present invention.

[0012]

The greatest characteristic of the present invention is that the mean diameter of a cell split by a network formed using a boride in the Co-Cr-Pt-B-group target is made so that it is fine and uniform. A cell which has been split by a networked formed of a boride greatly affects the

degree of dispersion of the target boride. This degree of dispersion of the boride affects the uniformity of the magnetic properties of the magnetic film when a magnetic film is being formed by sputtering using the target.

[0013]

Therefore, the Co-Cr-Pt-B-group target in the present invention makes the mean diameter of the cell split by a network formed of boride fine and uniform. As a result, the dispersions in the magnetic properties such as holding power and angle type ratio of the magnetic film are inhibited and the magnetic recording media can manufactured stably. The mean diameter of the cell split by a network formed of boride is restricted to 200 μm because when the mean diameter of the cell exceeds 200 μm , the uniformity of the magnetic properties of the magnetic film deteriorates conspicuously.

[0014]

There is a method which accelerates the cooling speed when the target is cast which can be used to make the mean diameter of the cell split by a network formed of boride in the Co-Cr-Pt-B-group target in the present invention fine and uniform.

[0015]

Since there is a great deal of supercooling during aggregation in alloys to which B has been added, fine equiaxed crystals are relatively easily formed by accelerating the cooling speed during casting. As a result, a cell which has been split by a network formed of boride are fine. Specific methods used to accelerate the cooling speed during casting are: a method which increases the cooling power of the casting mold itself; a method which makes the ingots thinner; a method which lowers the casting temperature and the like. However, a method which involves making the casting mold of a material which has a high cooling capacity such as copper and the like and a method which involves water-cooling the casting mold itself may also be used. By using these methods, the mean diameter of a cell split by a network made of a boride can be made finer more easily. In addition, the environment where the melting and casting are carried out also affects the cooling speed to a certain degree. However, these should be carried out in a vacuum where the gas constituent of the oxygen and the like in the target is reduced.

[0016]

It is also suitable as the dispersions in the magnetic properties such as the holding power and the angle type ratio of the magnetic film on the Co-Cr-Pt-B-group sputtered film can be reduced by carrying out hot rolling plastic processing such as hot-rolling and hot forging on the target material where the mean diameter of the cell split by a network formed by boride is 200 μm and under in the Co-Cr-Pt-B-group target, recrystallizing the matrix and making the mean crystal particle size of the matrix 40 μm and under.

[0017]

The method used to find the mean diameter and the matrix mean particle size of the cell split by a network formed of boride may involve observing the microtexture of the surface part and the center part of the target manufactured using an optical microscope from the sputtering side direction and measuring using the cutting method based on the photograph observed.

[0018]

In addition, the fact that there are dispersions in the mean diameter of the cell split by a networked formed of boride in the Co-Cr-Pt-B-group target in the present

invention and dispersions in the mean crystal particle size of the matrix is caused by dispersions in the film forming properties and particularly by changes over time when the sputtered film is made. As a result, the dispersions in the mean diameter of the network formed of boride should be inhibited insofar as possible during sputtering by making it 0.5 to 2.0 at as/aM. The dispersions in the mean crystal particle size of the matrix may be bs/bM in order to obtain a greater effect.

[0019]

Targets having few dispersions in the mean diameter of the cell split by a network formed of boride can accelerate the cooling during casting and the ingots can be made thinner by uniformly accelerating the cooling.

[0020]

In addition, targets having few dispersions in the mean crystal particle size of the matrix can be manufactured by controlling the hot rolling plastic processing conditions for the processing rate during hot plastic processing. Specifically, when the processing rate is too high, the dispersions caused by the anisotropy during hot plastic processing increase. When the processing

rate is too low, recrystallization occurs so that there is no hot plastic processing effect. In addition, when hot rolling is carried out as hot plastic processing, the anisotropy of the texture does not manifest greatly due to the rolling carried out by cross rolling so that it is recommended. In addition, heat treatment may be carried out before or after the hot plastic processing and texture control can be carried out.

[0021]

A suitable texture for the Co-Cr-Pt-B-group target in the present invention is $1 \leq B \leq 5$ at %, $5 \leq Pt \leq 30$ at %, $10 \leq Cr \leq 30$ at % with the residue essentially made up of Co. Next, we shall provide a more detailed explanation of the restrictions on each of the elements to be added.

[0022]

B has the effect of segregating to the grain boundary in the film and segregating the Pt element inside the grain. It also has the effect of segregating the Cr and other non-magnetic elements to the grain boundary. These effects are striking clear when 1 at % and above is added. In addition, B is an element which promotes the process of

becoming amorphous. When more than 15 at % is added, the crystallinity of the film is lost and it deteriorates the magnetic properties of the film so that $1 \leq B \leq 15$ at % is preferred.

[0023]

Pt increases the magnetic anisotropy by making a solution in the Co and has the effect of increasing the holding power of the film. A striking effect was seen when

/4

5 at % and above was added to increase the holding power. In addition, adding more than 30 at % significantly reduced the magnetic properties of the magnetic anisotropy which is an innate property of Co so that $5 \leq Pt \leq 30$ at % is preferable.

[0024]

Cr has the action of segregating to the grain boundary in the film and making the grain boundary non-magnetic so that the ferromagnetic Co grains were segmented magnetically. When less than 10 at % is added, the magnetic segmentation is insufficient. In addition, when more than 30 at % is added, the magnetism of the film itself is lowered too much so that $10 \leq CR \leq 30$ at % is preferable.

[0025]

Ti, Zr, Hf, V, Nb, Ta, Mo, W, Mn, Re, Ru, Os, Rh, Ir, Ni, Pd, Cu, Ag, Au and C can be added as elements added to improve the magnetic properties. Although these elements have an effect when small amounts are added, when the total amount exceeds 40 at %, the magnetic properties and the crystallinity are noticeably impaired. As a result, $0 < (Ti + Zr + Hf + V + Nb + Ta + Mo + W + Mn + Re + Ru + Os + Rh + Ir + Ni + Pd + Cu + Ag + Au + C) < 40$ at %.

[0026]

[Practical Embodiments]

(Practical Embodiment 1)

We produced a target measuring ϕ 101 mm x 5 mm^t having a composition of Co-20 Cr-10 Pt-5 B (at %) under the casting and hot rolling conditions indicated in Table 1. We collected test materials (hereinafter, "TP") from the sputtered surface part and the center part in the thickness direction of the target produced and used an optical microscope to observe the microtextures from the TP from the sputtering surface direction. Results of measuring the mean diameter of the cell split by a network formed of boride as well as the mean particle size of the matrix are

indicated in Table 2. However, it should be noted that the mean diameter of the cell split by a network formed by boride and the mean diameter of the matrix were the mean value of the respective surface part and center part.

[0027]

In addition, the matrix of the target which had not been hot rolled was not recrystallized and was configured of microcrystals. As a result, rigorous measurement of the mean particle size was difficult because of the optical microscope so that it could not be measured. A typical texture for the target in the present invention is indicated in the microtexture photographs of Sample 5 and Sample 10 indicated respectively in Table 1 and Table 2. It was found that by controlling the material for the casting mold and the thickness of the ingots as seen in Table 1, Table 2, Figure 1 and Figure 2, the mean diameter of the cell in the network formed of boride could be made fine and uniform. Furthermore, the ceramics indicated in Samples 11, 12 in Table 1 are casting molds used when casting using the lost wax method.

[0028]

[Table 1]

試料	鋳型材質	インゴット厚 (mm)	圧延率 (%)
1	Cu	25	25
2	Cu	30	25
3	Cu	35	25
4	Cu	40	0
5	Cu	40	25
6	Cu	40	50
7	Fe	25	25
8	Fe	35	0
9	Fe	35	25
10	Fe	40	25
11	セラミックス	25	25
12	セラミックス	40	25

[Captions:

1: Sample; 2: Casting Mold Material; 3: Ingot thickness (mm); 4: Rolling Rate (%); 5: Ceramic.

[0029]

[Table 2]

試料	セル 平均径 (μm)	表面部 セル 平均径 (μm)	中心部 セル 平均径 (μm)	表面／中心 セル平均径 比率	備考
1	2.7	2.4	3.0	0.80	本発明例
2	3.5	3.1	3.9	0.79	本発明例
3	4.1	3.4	4.8	0.71	本発明例
4	5.3	4.0	6.5	0.62	本発明例
5	5.3	4.1	6.5	0.63	本発明例
6	5.7	4.4	6.9	0.64	本発明例
7	8.3	6.7	9.8	0.68	本発明例
8	10.1	7.0	13.2	0.53	本発明例
9	10.6	7.3	13.8	0.53	本発明例
10	12.5	7.9	17.0	0.46	本発明例
11	21.4	15.0	27.8	0.54	比較例
12	23.0	15.9	30.1	0.53	比較例

[Captions:

1: Sample; 2: Mean Diameter of Cell (μm); 3: Surface Part Cell Mean Diameter (μm); 4: Center Part Cell Mean Diameter (μm) 5: Surface/Center Cell Mean Diameter Ratio; 6: Remarks; 7: Practical Embodiment of Present Invention; 8: Comparative Example of Present Invention.

[0030]

/5

Table 3

試料	マトリクス 平均粒径 (μm)	表面部 マトリクス 平均粒径 (μm)	中心部 マトリクス 平均粒径 (μm)	表面/中心 マトリクス 平均粒径 比率	備考
1	9	8	9	0. 89	本発明例
2	11	10	12	0. 83	本発明例
3	12	11	12	0. 92	本発明例
4	—	—	—	—	本発明例
5	12	10	13	0. 77	本発明例
6	11	8	14	0. 57	本発明例
7	16	15	17	0. 88	本発明例
8	—	—	—	—	本発明例
9	17	16	17	0. 94	本発明例
10	19	18	20	0. 90	本発明例
11	18	17	19	0. 89	比較例
12	23	21	25	0. 84	比較例

[captions:

1: Sample; 2: Mean Particle Size of Matrix (μm); 3: Surface Part, Mean Particle Size of Matrix (μm); 4: Center Part, Mean Particle Size of Matrix (μm); 5: Surface/ Center Part Mean Particle Size of Matrix (%); 6: Remarks; 7: Practical Embodiment of the Present Invention; 8: Comparative Embodiment

[0031]

(Practical Embodiment 2) We formed a Cr background film at conditions of substrate temperature of 150°C, Ar pressure of 0.66 Pa and DC electric power of 500 W and a

magnetic film using a variety of targets of Co-20 Cr-10 Pt-5 B (at %) produced at the conditions indicated in Table 1 on a substrate using an Al substrate which had been subjected to NiP plating.

[0032]

We made a film forming substrate where the overall film forming time ranges from 1 hour to 5 hours in 1 hour intervals in order to study the dispersions in the magnetic films and indicated the results of measuring the magnetic holding power H_c measured using a VSM (vibration sample magnetometer) in Table 4. However, it should be noted that Table 4 indicates the magnetic holding force when the film forming time for each of the samples for 1 hour is 100. It can be seen from Table 4 that the film properties during sputtering film formation was stabilized by making the mean diameter of the cell of the network formed by a boride as well as the mean crystal particle size of the matrix fine and uniform.

[0033]

[Table 4]

試料	保磁力 (相対値)					備考
	1時間	2時間	3時間	4時間	5時間	
1	100	100	100	99	99	本発明例
2	100	101	99	100	99	本発明例
3	100	101	102	100	100	本発明例
4	100	104	95	94	94	本発明例
5	100	98	101	102	97	本発明例
6	100	97	102	98	97	本発明例
7	100	100	103	96	97	本発明例
8	100	94	95	98	90	本発明例
9	100	102	98	98	93	本発明例
10	100	102	98	90	93	本発明例
11	100	90	91	101	87	比較例
12	100	98	102	89	88	比較例

[captions:

1: Samples; 2: Magnetic Holding Force (relative value); 3: 1 hour; 4: 2 hours; 5: 3 hours; 6: 4 hours; 7: 5 hours; 8: Remarks; 9: Practical Embodiment of the Present Invention; 10: Comparative Embodiment

[0034]

(Practical Embodiment 3) We formed a target measuring ϕ 101 mm x 5 mm having a composition to which Ta, Zr, Cu, Mo, Ti and Ni had been added at 2 at % respectively individually to raw materials of Co-20 Cr-10 Pt-5B (at %) under the casting conditions and hot rolling condition indicated in Table 5. We observed the microtexture of the center part of the target made from the sputtering surface direction.

Results of measuring the mean diameter of the cell split by a network formed of a boride and the mean particle size of the matrix using the cutting method are indicated in Table 6. It can be seen from Table 6 that the mean diameter of the cell split by a network formed of a boride can be made fine.

[0035]

[Table 5]

/6

Production Method	Casting Material	Ingot Thickness (mm)	Rolling Rate (%)
A	Cu	30	25
B	Cu	40	25
C	Ceramic	40	25

[0036]

[Table 6]

* * 【表6】

試料	添加元素	作製方法	セル平均径 (μm)	マトリクス 平均粒径 (μm)	備考
21	Ta	A	35	10	本発明例
22	Ta	B	60	11	本発明例
23	Ta	C	280	19	比較例
24	Zr	A	30	11	本発明例
25	Zr	B	55	11	本発明例
26	Zr	C	270	13	比較例
27	Cu	A	45	13	本発明例
28	Cu	B	65	13	本発明例
29	Cu	C	310	22	比較例
30	Mo	A	40	12	本発明例
31	Mo	B	70	14	本発明例
32	Mo	C	820	19	比較例
33	Ti	A	35	11	本発明例
34	Ti	B	65	12	本発明例
35	Ti	C	290	20	比較例
36	Ni	A	45	14	本発明例
37	Ni	B	70	15	本発明例
38	Ni	C	330	21	比較例

[captions:

1: Sample; 2: Element Added; 3: Production Method; 4: Mean Cell Diameter (μm); 5: Matrix Mean Particle Size (μm); 6: Remarks; 7: Practical Embodiment of the Present Invention; 8: Comparative Embodiment.

[0037]

[Effect of Invention]

The present invention provides a Co-Cr-Pt-B-group target which inhibits dispersions in the magnetic properties of a Co-Cr-Pt-B-group of a magnetic recording medium for magnetic disk devices and the like and is a technique which is indispensable for manufacturing magnetic recording media.

[Brief Explanation of Figures]

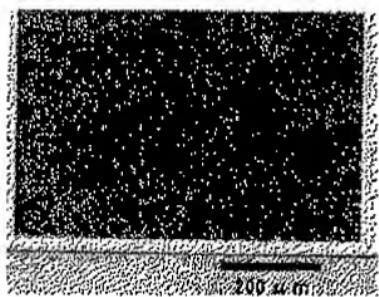
[Figure 1] An optical microphotograph indicating the microtexture observing the target material for Sample 5 in Table 1 indicating the practical embodiment of the Co-Cr-Pt-B-group target in the present invention seen from the sputtering direction.

[Figure 2] An optical microphotograph indicating the microtexture of the target material of Sample 10 in Table 1 indicating a practical embodiment of the Co-Cr-Pt-B-group target in the present invention seen from the sputtering direction.

[Figure 1]



[Figure 2]



[Amendment of the Proceedings]

[Date of Filing] March 5, 2001

[Amendment 1]

[Document to be Amended] Specification

[Name of Document to be Amendment] 0018

[Method of Amendment] Change

[Details of Amendment]

[0018]

In addition, the fact that there are dispersions in the mean diameter of the cell split by a network formed of a boride as well as the mean crystal particle size in the Co-Cr-Pt-B-group target in the present invention and the fact that the dispersions in the film properties particularly the dispersions in the mean diameter in the network formed by a boride are caused by changes over time when the film is formed by sputtering are such that it should be possible to inhibit the changes over time when sputtering is carried out when this is 0.5 to 2.0 at as/aM and the dispersions in the mean crystal particle size of the matrix in order to obtain the effect should be placed at 0.5 to 2.0 at bs/bM.